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J.A.KEMP &amp; CO.

BY HAND

14 SOUTH SQUARE, GRAY'S INN  
LONDON WC1R 5JJ

International Preliminary Examining Authority  
The European Patent Office  
Erhardstrasse 27  
D-80331 Munich  
Germany

TELEPHONE: + 44 20 7405 3292  
FACSIMILE: + 44 20 7242 8932  
E-MAIL: mail@jukemp.com  
WEBSITE: www.jakemp.com

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Dear Sirs

International Patent Application No. PCT/GB2004/001353

METAL PIGMENT COMPOSITION

Our Ref: N.88299A TAC/JAT/ejb

We refer to the Communication of 19 July 2004 concerning the Written Opinion on the above application. The applicant wishes to file amendments under Article 34 PCT. Please replace claims 1 to 28 with new claims 1 to 31 enclosed, corresponding to replacement pages 26 to 30. For ease of reference, original claims 1 to 28, showing the amendments in manuscript, are also enclosed.

The claims have been amended to limit the milling fluid to those that are both solvent and water miscible. Basis for this amendment can be found on page 3, line 31 to page 4, line 2 of the description. Claim 1 has been further limited such that the milling fluid does not comprise alcohols or esters. These two values have been deleted from the list of milling fluids exemplified at page 6, lines 1 to 3. New claim 2 limits the mixture milled such that it consists of metal powder and a milling fluid consisting of an alcohol or an ester. Basis can be found at page 3, lines 9 to 10, as well as in the list on page 6, lines 1 to 3. The remaining claims have been amended for consistency with the new independent claims.

Document D1 cited by the Examiner refers to milling in an inert medium comprising transesterified alkyl esters of vegetable oils. Such substances are immiscible with water, and hence do not satisfy the condition of the present invention that the milling fluid is both solvent and water miscible. The present claims are therefore novel over D1.

D2 is not relevant to the present application, since it is directed towards pre-prepared metal particles, and does not describe a process of using a milling fluid in milling a metal powder, as required in the present invention.

OXFORD OFFICE · MAGDALEN CENTRE · THE OXFORD SCIENCE PARK · OXFORD OX4 4GA  
TELEPHONE: + 44 1865 784760 · FACSIMILE: + 44 1865 784775

MUNICH OFFICE · BAYER KAHRE · BAYERSTRASSE 83 · D-80335 MÜNCHEN · GERMANY  
TELEPHONE: + 49 89 24 22 97 340 · FACSIMILE: + 49 89 24 22 97 350

A M SENIOR, MA, EPA.CPA.  
S BENTHAM, MA, EPA.CPA.  
M L S AYERS, DSC, EPA.CPA.  
G C WOODS, MA, EPA.CPA.  
T A CRESSWELL, BSC, EPA.CPA.  
M A MARSHALL, BSC, EPA.CPA.<sup>\*\*</sup>  
A J WERR, MA, EPA.CPA.  
M J NICHOLLS, MA, EPA.CPA.  
N J K PRICH, BSC, EPA.CPA.  
C M KEEN, MA, EPA.CPA.  
J C LEMMING, MA, EPA.CPA.  
S L SMITH, MA, EPA.CPA.  
I F RINSON, BSC, EPA.CPA.

DR. T J DUCKWORTH, EPA.CPA.  
S M WRIGHT, BSC, EPA.CPA.<sup>\*\*</sup>  
P J H CAMPBELL, MA, EPA.CPA.  
J H SEXTON, BSC, EPA.CPA.<sup>\*\*</sup>  
G W MCCCLUSKIE, BSC, EPA.CPA.<sup>\*\*</sup>  
C H MERRYWEATHER, BA, EPA.CPA.  
S E KOOLES, MA, EPA.CPA.<sup>\*\*</sup>  
DR. A J DUCKETT, EPA.CPA.  
A BENTHAM, MA, EPA.CPA.  
DR. R P TYSON, EPA.CPA.  
R C SRINIVASAN, MA, EPA.CPA.  
J A FISH, LLB, ILM, EPA.CPA.  
R I BARLOW, BSC, EPA.CPA.

ASSOCIATES :-  
DR. S ALI, EPA.CPA.  
M P ROBERTS, MENG, EPA.CPA.  
DR. M C CHADWICK, EPA.  
A L SIMONS, MChem, EPA.CPA.  
DR. P M TUSWORTH, EPA.CPA.  
K M FITCHELL, LLB.<sup>\*\*</sup>  
C J CROWE, BA.<sup>\*\*</sup>  
J M KEOGH, BEng.<sup>\*\*</sup>  
DR. M P JACKSON, EPA.CPA.  
DR. D'POWER, EPA.CPA.  
T J B HOPKIN, MA, MEng, EPA.CPA.  
DR. H FORBES, EPA.CPA.

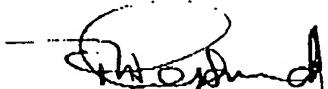
K A J BUTTON, MA, MPhil, EPA.CPA.  
S A GORSUCH, MA, EPA.CPA.  
DR. S A RAYNER, EPA.  
E L ELWELL, MChem, MSc, EPA.  
CONSULTANTS :-  
DR. R P FAWCETT, EPA.CPA.  
D M GOLDIN, BSC, EPA.CPA.  
P G A ELLIS-JONES, MA, EPA.CPA.<sup>\*\*</sup>  
Solicitor - ATTMA  
European Trade Mark Representative  
Registered Australian Patent & Trade  
Mark Attorney

Claim 10 of D3 mentions alcohols and esters as a solvent for the crushing aid/binding agent employed in the process. Present claim 1 is limited to exclude alcohols and esters, and hence D3 does not anticipate this claim. Claim 2 has been limited such that when alcohols and esters are used, no other substance, such as a crushing aid, is present in the mixture. D3 does not mention use of the solvent independently of the binding agent/crushing aid in milling metal powders. The present claims are therefore novel over D3.

As described at page 3, lines 29 to 31 of the present description, it is an object of the present invention to provide a "universal" pigment composition, i.e. a composition that is suitable for both water and solvent based coatings. D1 does not describe fluids that are both solvent and water miscible, D2 relates to treated metal particles for aqueous coating systems, and D3 is directed towards odour-free coatings. It would not therefore be obvious from the prior art to arrive at the present process as the solution to this problem, and the present invention possesses an inventive step over the cited documents.

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Yours faithfully



T A CRESSWELL  
AUTHORISED REPRESENTATIVE

REPLACEMENT SHEET

CLAIMS

1. Process for producing a low volatility metal flake pigment composition, which comprises milling metal powder in a milling fluid, wherein the milling fluid comprises a non-aqueous, non-hydrocarbon, low volatility fluid that is both solvent and water miscible, other than alcohols or esters.
2. Process for producing a low volatility metal flake pigment composition, which comprises milling a mixture consisting of metal powder and a non-aqueous, non-hydrocarbon, low volatility milling fluid that is both solvent and water miscible, wherein the milling fluid consists of an alcohol or an ester.
3. Process according to claim 1, which further comprises the addition of one or more substances that act as a lubricant and/or corrosion inhibitor.
4. Process according to any of the preceding claims, which further comprises the step of removing oversize or undersize particles after milling.
5. Process according to any of the preceding claims, which further comprises concentrating after milling to a metal flake pigment paste of 50-90% metal by weight.
6. Process according to claim 5, wherein the paste is converted to a granule form by incorporating an organic binder to form a coherent paste of organic binder, milling fluid and metal flake pigment.
7. Process according to claim 6, wherein the paste of organic binder, milling fluid and metal flake pigment is in a particulate form such that at least 98% by weight of the particles are retained on a sieve having a 150 $\mu$ m aperture.

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8. Process according to claim 6 or claim 7, wherein the granules have a length of 5 to 20mm and a thickness of 1.5 to 3mm.
9. Process according to any one of claims 6 to 8 wherein part or all of the milling fluid is removed at elevated temperature.
10. Process according to any of the preceding claims, which further comprises a step of treating the milled metal flakes in the milling fluid.
11. Process according to claim 10, wherein the metal flakes are treated with a phosphate, silica or alumina, ammonium dichromate or ammonium or potassium permanganate.
12. Process according to any of the preceding claims, wherein the metal flakes are thermally treated after the milling step.
13. Process according to claim 1 or any one of claims 3 to 12, wherein the milling fluid is ethylene glycol, dimethylene glycol, diethylene glycol, trimethylene glycol, triethylene glycol, propylene glycols, butylene glycols, glycerol, gamma butyrolactone, 2-pyrrolidone, N-methyl pyrrolidone, isophorone, triacetin, 2,5-hexanedione, tetraethylene pentamine, triethyl phosphate, ethyl acetoacetate, n-methyl formamide, propylene carbonate, ethylene glycol monobutyl ether, diethylene glycol monomethyl, monoethyl, monopropyl and monobutyl ethers, triethylene glycol monomethyl, monoethyl, monopropyl and monobutyl ethers, diethylene glycol dimethyl, diethyl, dipropyl and dibutyl ethers, triethylene glycol dimethyl, diethyl, dipropyl and dibutyl ethers, propylene glycol monobutyl ether, dipropylene glycol monomethyl, monoethyl, monopropyl and monobutyl ethers, tripropylene glycol monomethyl, monoethyl, monopropyl and monobutyl ethers, dipropylene glycol dimethyl, diethyl, dipropyl and dibutyl

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ethers, tripropylene glycol dimethyl, diethyl, dipropyl and dibutyl ethers, diamyl ether, or a mixture of any two or more of these.

14. Process according to claim 13, wherein the milling fluid is propylene carbonate, tripropylene glycol monomethyl ether, dipropylene glycol dimethyl ether or dipropylene glycol n-propyl ether.

15. Process according to claim 2 or any one of claims 4 to 12, wherein the milling fluid consists of pentyl alcohol, hexyl alcohol, 2(2-n-butoxy ethoxy ethanol), tetrahydropyran, tetrahydropyran-2-methanol, methoxypropyl acetate, ethylene glycol monobutyl ether acetate, diethylene glycol monomethyl ether acetate, diethylene glycol monoethyl ether acetate, diethylene glycol monobutyl ether acetate, dipropylene glycol monomethyl ether acetate, propylene glycol diacetate, methyl, ethyl and propyl lactate or the monomethyl, monoethyl, dimethyl and diethyl esters of succinic, glutaric and adipic acids, or a mixture of any two or more of these.

16. Process according to claim 15, wherein the milling fluid is diethylene glycol monomethyl ether acetate, diethylene glycol monobutyl ether acetate, or mixtures of dimethyl esters of adipic, glutaric and succinic acids.

17. Process according to any of the preceding claims, wherein the metal is aluminium, zinc, copper, tin, nickel, silver, gold, iron, or an alloy thereof.

18. Process according to claim 17, wherein the metal is aluminium or gold bronze.

19. Process according to any of the preceding claims, wherein the metal pigment particles have a median diameter of 6 $\mu$ m to 600 $\mu$ m.

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20. Process according to claim 19, wherein the metal pigment particles have a median diameter of 10µm to 300µm.
21. Process according to any of claims 3 to 20, wherein the corrosion inhibitor is calcium phosphate, magnesium phosphate, calcium phosphosilicate, calcium strontium phosphosilicate, aluminium zirconium zinc phosphosilicate, calcium strontium zinc phosphosilicate, alkyl and dialkyl phosphates, phosphites and their derivatives, phosphonic acid derivatives, phosphate esters of long chain ethoxylated alcohols, organic silanes and silicates, compounds of molybdenum, vanadium, titanium, zirconium, and tungsten and heteropolyanionic compounds thereof, ammonium dichromate or chromic acid.
22. Process according to any of claims 3 to 21, wherein the lubricant is a long chain or polymeric fatty acid, a phosphate ester of a long chain ethoxylated alcohol, lauryl phosphonic acid, lauryl phosphate or a mineral oil.
23. Process according to any of the preceding claims, comprising ball milling the metal powder.
24. Use of a non-aqueous, non-hydrocarbon milling fluid in a process of milling a metal powder as claimed in any of the preceding claims.
25. A metal pigment paste obtainable by the process of any of claims 1 to 23.
26. A granule obtainable by the process of any of claims 6 to 20.
27. A granule produced by the process of any of claims 6 to 20.
28. An ink or surface coating comprising the metal pigment paste or

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granules produced by a process according to any of claims 1 to 23 and surface coating binders dissolved or dispersed in water, solvent or mixtures of the two.

29. Article obtainable by shaping a composition comprising granules produced by the process of any of claims 6 to 20.

30. Article obtainable by injection moulding or by extrusion of a thermoplastic, comprising granules produced by the process of any of claims 6 to 20.

31. Shaped article comprising a thermoplastic or thermosetting polymer and granules produced by the process of any of claims 6 to 20.